FORM PTO-1390 (REV. 10-95) U.S. DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE	ATTORNEY'S DOCKET NUMBER
TRANSMITTAL LETTER TO THE UNITED STATES DESIGNATED/ELECTED OFFICE (DO/EO/US) CONCERNING A FILING UNDER 35 U.S.C. 371	**7713M/DQ**
	U.S. APPLICATION NO. (if known, see 37 CFR 1.5) **09/787852**

INTERNATIONAL APPLICATION NO. PCT/US99/22396	INTERNATIONAL FILING DATE 24 September 1999	PRIORITY DATE CLAIMED 25 September 1998

TITLE OF INVENTION
Granular Detergent Compositions Having Improved Solubility Profiles

APPLICANT(S) FOR DO/EO/US
CAPECI, Scott William et al.

Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information.

1. [x] This is a FIRST submission of items concerning a filing under 35 U.S.C. 371.

2. [] This is a SECOND or SUBSEQUENT submission of items concerning a filing under 35 U.S.C. 371.

3. [] This express request to begin national examination procedures (35 U.S.C. 371(f) at any time rather than delay examination until the expiration of the applicable time limit set in 35 U.S.C. 371(b) and PCT Articles 22 and 39(l).

4. [x] A proper Demand for International Preliminary Examination was made by the 19th month from the earliest claimed priority date.

5. [x] A copy of the International Application was filed (35 U.S.C. 371(c)(2))

    a. [] is transmitted herewith (required only if not transmitted by the International Bureau).

    b. [] has been transmitted by the International Bureau.

    c. [x] is not required, as the application was filed in the United States Receiving Office (RO/US).

6. [] A translation of the International Application into English (35 U.S.C. 371(c)(2)).

7. [x] Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371(c)(3))

    a. [] are transmitted herewith (required only if not transmitted by the International Bureau).

    b. [] have been transmitted by the International Bureau.

    c. [] have not been made; however, the time limit for making such amendments has NOT expired.

    d. [x] have not been made and will not be made.

8. [] A translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371(c)(3)).

9. [] An oath or declaration of the inventor(s) (35 U.S.C. 371(c)(4)).

10. [] A translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371(c)(5)).

Items 11. to 16. below concern document(s) or information included:

11. [] An Information Disclosure Statement under 37 CFR 1.97 and 1.98.

12. [] An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.

13. [x] A FIRST preliminary amendment.

    [] A SECOND or SUBSEQUENT preliminary amendment.

14. [] A substitute specification.

15. [x] A change of power of attorney and/or address letter.

16. [] Other items or information:

"Express Mail" mailing label number _EL483620252US_

Date of Deposit _22 March 2001_

I hereby certify that this paper/fee is being deposited with the United States Postal Service "Express Mail Post Office to Addressee" service under 37 CFR 1.10 on the date indicated above and is addressed to The Assistant Commissioner of Patents, Washington, D C 20231

Administrator-Mailing Application

Signature _Virginia C Byrd_

U.S. APPLICATION NO. (If known, see 37 CFR 1.5)	INTERNATIONAL APPLICATION NO.	ATTORNEY'S DOCKET NUMBER
09/787853	PCT/US99/22396	7713M/DQ

	CALCULATIONS	PTO USE ONLY

ENTER APPROPRIATE BASIC FEE AMOUNT =	**$710**	
Surcharge of $130.00 for furnishing the oath or declaration later than [ ] 20 [ ] 30 months from the earliest claimed priority date (37 CFR 1.492(e)).	$0	

CLAIMS	NUMBER FILED	NUMBER EXTRA	RATE		
Total Claims	8-20 =	0	x $18.00	$0	
Independent Claims	1-3 =	0	x $80.00	$0	
MULTIPLE DEPENDENT CLAIM(S) (if applicable)			$270.00	$0	
TOTAL OF ABOVE CALCULATIONS =				**$710**	
Processing fee of $130.00 for furnishing the English translation later than [ ] 20 [ ] 30 months from the earliest claimed priority date (37 CFR 1.492(f)).				$0	
TOTAL NATIONAL FEE =				**$710**	
Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be accompanied by an appropriate cover sheet (37 CFR 3.28,3.31). $40.00 per property +				$0	
TOTAL FEES ENCLOSED =				**$710**	
				Amount to be refunded	$
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a. [ ] A check in the amount of $ _____ to cover the above fees is enclosed.

b. [x] Please charge my Deposit Account No. __16-2480__ in the amount of $ _710_ to cover the above fees. A duplicate copy of this sheet is enclosed.

c. [x] The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment to Deposit Account No. _16-2480_ . A duplicate copy of this sheet is enclosed.

NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR 1.137(a) or (b)) must be filed and granted to restore the application to pending status.

SEND ALL CORRESPONDENCE TO:

M. Dressman, Patent Attorney Patent Division – 5W6 - Box 492 The Procter & Gamble Company Ivorydale Technical Center 5299 Spring Grove Avenue Cincinnati, Ohio 45217-1087	_____ Signature T. David Reed Name 32,931 Registration Number

Case 7713M

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In the Matter of:

U.S. National Phase Entry

Under 35 USC § 371 from

the International Application of   :

CAPECI, Scott William et al.   :

Int'l Application No. PCT/US99/22396   :

Filed in the RO/US on 24 September 1999   :

Entitled: Granular Detergent Compositions Having Improved
                 Solubility Profiles

PRELIMINARY AMENDMENT UNDER 37 CFR § 1.112

Assistant Commissioner for Patents

Washington, D.C. 20231

Dear Sir:

　　　　Prior to Examination and computation of the fees for entering the captioned International Application into the U.S. National Phase, please preliminarily amend the above-identified application as follows and consider the following Remarks.

AMENDMENTS

IN THE CLAIMS

　　　　Please amend Claims as follows:

8.　　　　(Amended) The composition as claimed in Claim 7 wherein said composition has insoluble residues and at least about 90%of said insoluble residues have a particle size of less than 15 μm.

REMARKS

Claims 1-7 remain in this application. Claim 8 has been amended by eliminating multiple dependent claims and deleting preferably clauses. Attached hereto is a marked-up version of the changes made to the claims by the current amendment. The attached page is captioned "Version With Markings to Show Changes Made".

The support for these amendments is found in the claims as originally filed. These amendments are being entered to bring the claims into conformance with, *inter alia*, 37 CFR §1.75; no new matter is added.

Respectfully submitted,

By _____

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19 March 2001
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In the claims:

1.   A granular detergent composition having an average bulk density of at least about 400 g/L and characterized by a rate of dispersion as defined by the equation:

$$R = R^* + (1 - R^*) \exp\left(-\left(\frac{t}{DT(t_{wash})}\right)^m\right)$$

where R is the residual undissolved detergent at any point in time, t, R* is the long term residual undispersed detergent having a value of less than about 14% of the total amount of an initial dosage of detergent, t is any single point in time, m is a stretching exponent having a value of less than about 2, DT is dispersion time having a value of less than about 0.5 and t_{wash} is the time of the wash cycle.

2.   The granular detergent composition as claimed in Claim 1 wherein at least 90% of the insoluble residues of the granular detergent composition have an average particle size of less than about 10 μm.

3.   The granular detergent composition as claimed in Claim 1 wherein R* has a value of less than about 7%, m has a value of less than about 1.5 and DT has a value of less than about 0.25.

4.   The granular detergent composition as claimed in Claim 3 wherein R* has a value of less than about 3.5%, m has a value of less than about 1 and DT has a value of less than about 0.12.

5.   The granular detergent composition as claimed in Claim 1 wherein said detergent composition has a rate of dissolution as defined by the equation:

$$U = U^* + (1 - U^*) \exp\left(-\left(\frac{t}{RT(t_{wash})}\right)^n\right)$$

where U is the fraction of undissolved surfactant at any point in time, t, U* is the long term surfactant residual undissolved surfactant having a value of less than about 14% of the total amount of an initial dosage of surfactant, t is any single point in time, n is a stretching exponent having a value of less than about 2, RT is dissolution time having a value of less than about 0.5 and t_{wash} is the time of the wash cycle.

6.   The granular detergent composition as claimed in Claim 4 wherein U* has a value of less than about 7%, n has a value of less than about 1.5 and RT has a value of less than about 0.25.

7.   The granular detergent composition as claimed in Claim 5 wherein U* has a value of less than about 3.5%, n has a value of less than about 1 and RT has a value of less than about 0.12.

8.   The composition as claimed in Claim 7 [any of claims 1-7] wherein said composition has insoluble residues and at least about 90%of said insoluble residues have a particle size of less than 15 μm.

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GRANULAR DETERGENT COMPOSITIONS HAVING IMPROVED SOLUBILITY PROFILES

Technical Field

The present invention relates to granular detergent compositions having improved solubility profiles and in particular, to granular detergent compositions that match or exceed the cleaning performance of liquid detergents while avoiding the negative attributes associated with granular products.

Background of the Invention

Recently, there has been considerable interest within the detergent industry for laundry detergents which have the convenience, aesthetics and solubility of liquid laundry detergent products, but retain the cleaning performance and cost of granular detergent products. The problems, however, associated with past granular detergent compositions with regard to aesthetics, solubility and user convenience are formidable. Such problems have been exacerbated by the advent of "compact" or low dosage granular detergent products which typically do not dissolve in washing solutions as well as their liquid laundry detergent counterparts. These low dosage detergents are currently in high demand as they conserve resources and can be sold in small packages which are more convenient for consumers prior to use, but less convenient upon dispensing into the washing machine as compared to liquid laundry detergent which can be simply poured directly from the bottle as opposed to "scooped" from the box and then dispensed into the washing solution.

As mentioned, such low dosage or "compact" detergent products unfortunately experience dissolution problems, especially in cold temperature laundering solutions (i.e., less than about 30°C). More specifically, poor dissolution results in the formation of "clumps" which appear as solid white masses remaining in the washing machine or on the laundered clothes after conventional washing cycles. These "clumps" are especially prevalent under cold temperature washing conditions and/or when the order of addition to the washing machine is laundry detergent first, clothes second and water last (commonly known as the "Reverse Order Of Addition" or "ROOA"). Such undesirable "clumps" are also formed if the consumer loads the washing machine in the order of clothes, detergent and then water. Similarly, this clumping phenomenon can contribute to the incomplete dispensing of detergent in washing machines

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equipped with dispenser drawers or in other dispensing devices, such as a granulette. In this case, the undesired result is undissolved detergent residue in the dispensing device.

It has been found that the cause of the aforementioned dissolution problem is associated with the "bridging" of a "gel-like" substance between surfactant-containing particles to form undesirable "clumps." The gel-like substance responsible for the undesirable "bridging" of particles into "clumps" originates from the partial dissolution of surfactant in the aqueous laundering solutions, wherein such partial dissolution causes the formation of a highly viscous surfactant phase or paste which binds or otherwise "bridges" other surfactant-containing particles together into "clumps." This undesirable dissolution phenomena is commonly referred to as "lump-gel" formation. In addition to the viscous surfactant "bridging" effect, inorganic salts have a tendency to hydrate which can also cause "bridging" of particles which linked together via hydration. In particular, inorganic salts hydrate with one another to form a cage structure which exhibits poor dissolution and ultimately ends up as a "clump" after the washing cycle. It would therefore be desirable to have a detergent composition which does not experience the dissolution problems identified above so as to result in improved cleaning performance.

The prior art is replete with disclosures addressing the dissolution problems associated with granular detergent compositions. For example, the prior art suggests limiting the use and manner of inorganic salts which can cause clumps via the "bridging" of hydrated salts during the laundering cycle. Specific ratios of selected inorganic salts are contemplated so as to minimize dissolution problems. Such a solution, however, constricts the formulation and process flexibility which are necessary for current commercialization of large-scale detergent products. Various other mechanisms have been suggested by the prior art, all of which involve formulation alteration, and thereby reduce formulation flexibility. As a consequence, it would therefore be desirable to have a detergent composition having improved dissolution without significantly inhibiting formulation flexibility.

Accordingly, the need remains to identify a mechanistic approach to the performance of granular detergent compositions over a broad range of conditions and for a granular composition that has improved dispersion and solubility over conventional granular detergent compositions.

Summary of the Invention

This need is met by the present invention wherein granular detergent compositions having particular dissolution and solubility profiles have been identified that provide cleaning performance on par with liquid detergent compositions while maintaining the formulation flexibility provided by granular admixes.

In accordance with the present invention, a granular detergent composition having an average bulk density of at least about 400 g/L and characterized by a rate of dispersion as defined by the equation:

$$R = R^* + (1 - R^*) \exp\left(-\left(\frac{t}{DT(t_{wash})}\right)^m\right)$$

where R is the residual undispersedv detergent at any point in time, t, R* is the long term residual undispersed detergent having a value of less than about 14% of the total amount of an initial dosage of detergent, t is any single point in time, m is a stretching exponent having a value of less than about 2, DT is dispersion time having a value of less than about 0.5 and t_{wash} is the time of the wash cycle. In preferred embodiments, at least 90% of the insoluble particulate residues of said granular detergent composition having a particles size of less than 15 μm are provided and/or the detergent composition has a rate of dissolution as defined by the equation:

$$U = U^* + (1 - U^*) \exp\left(-\left(\frac{t}{RT(t_{wash})}\right)^n\right)$$

where U is the fraction of undissolved surfactant at any point in time, t, U* is the long term surfactant residual undissolved surfactant having a value of less than about 14% of the total amount of an initial dosage of surfactant, t is any single point in time, n is a stretching exponent having a value of less than about 2, RT is dissolution time having a value of less than about 0.5 and t_{wash} is the time of the wash cycle.

Detailed Description of the Preferred Embodiments

Definitions

As used herein, the word "particles" means the entire size range of a detergent final product or component or the entire size range of discrete particles, agglomerates, or granules in a final detergent product or component admixture. It specifically does not refer to a size fraction (i.e., representing less than 100% of the entire size range) of any of these types of particles

unless the size fraction represents 100% of a discrete particle in an admixture of particles. For each type of particle component in an admixture, the entire size range of discrete particles of that type have the same or substantially similar composition regardless of whether the particles are in contact with other particles. For agglomerated components, the agglomerates themselves are considered as discrete particles and each discrete particle may be comprised of a composite of smaller primary particles and binder compositions. As used herein, the phrase "geometric mean particle diameter" means the geometric mass median diameter of a set of discrete particles as measured by any standard mass-based particle size measurement technique, preferably by dry sieving. As used herein, the phrase "geometric standard deviation" or "span" of a particle size distribution means the geometric breadth of the best-fitted log-normal function to the above-mentioned particle size data which can be accomplished by the ratio of the diameter of the 84.13 percentile divided by the diameter of the 50th percentile of the cumulative distribution ($D_{84.13}/D_{50}$); See Gotoh et al, *Powder Technology Handbook*, pp. 6-11, Meral Dekker 1997.

As used herein, the phrase "builder" means any inorganic material having "builder" performance in the detergency context, and specifically, organic or inorganic material capable of removing water hardness from washing solutions. As used herein, the term "bulk density" refers to the uncompressed, untapped powder bulk density, as measured by pouring an excess of powder sample through a funnel into a smooth metal vessel (e.g., a 500 ml volume cylinder), scraping off the excess from the heap above the rim of the vessel, measuring the remaining mass of powder and dividing the mass by the volume of the vessel.

The granular detergents of the present invention satisfy the desired performance characteristics of solubility and dispersion via the optimal selection of a series of parameters that allow the design of superior performing granular detergents.

While not wishing to be bound by theory, a series of transitions may be employed to describe the solubility of a granular detergent from the point in time at which the powder is first wetted by the wash solution to the point in time at which the powder as reach its end point of dissolution into the wash solution. The series of transitions starts with the rate of dispersion of the bulk mass of the powder, typically made up of millions of particles per dose, into well-dispersed single particles within the wash solution. Once dispersed, the individual particles are free to dissolve. Thus, proper dispersion of particles is important to the design of a superior performing powdered detergent. On the other hand, a poor dispersion of powder will tend to

form the aforementioned lump-gels that are slow to dissolve and prone to leave consumer undesirable residues on fabrics.

The second of the transitions which may be employed to describe the solubility of powdered detergent is the rate of dissolution of the particles into the wash solution. Dissolution of dispersed particles continues after dispersion of the granules is complete. The key parameter of a superior performing granular detergent is that the well-dispersed particles dissolve relatively quickly as compared to the length of the wash cycle. The earlier in time during the wash cycle that the powdered detergent is dissolved, the earlier detergent active ingredients are delivered to the wash providing a longer exposure of soiled fabrics to cleaning agents and providing for improved cleaning performance.

The last of the transitions employed to describe the solubility of the powdered detergent, is the amount of powder which will not dissolve given a reasonable length of time in which to do it, otherwise known as insoluble particles. Insoluble particles result in visible residues on fabrics as these insoluble particles are filtered from the wash solution by the fabrics themselves. Minimization of the particle size of these insoluble particles present in a powdered detergent leads to improve performance from high levels of active agents in the wash solution and to higher consumer acceptance from fewer visible residues on fabrics.

Each of the transitions, from rate of dispersion to rate of dissolution to insoluble residues, may be described by a parameter or inter-related parameters which together identify a superior performing detergent composition. However, it is the rate of dispersion and the amount of insoluble particles remaining which are the most important of factors for gauging detergent performance as they are linked to consumer recognizable impacts such as residues on clothing and how quickly the bulk mass of added detergent dissipates from view.

Rate of Dispersion

The rate of dispersion as employed herein is a measure of the speed at which particles disperse throughout a wash solution. The rate of dispersion is a complex phenomena that is due in large part to interparticle interaction. However, this phenomena may be accurately described via the general stretched-exponential equation:

$$R = R^* + (1 - R^*) \exp\left(-\left(\frac{t}{DT\left(t_{wash}\right)} \right)^m \right)$$

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where R is the residual at any point in time, t, R* is the long term residual, i.e. undispersed particles, t is any single point in time, m is a stretching exponent that describes how the powder acts at the initial point in time, i.e the effect of initial wetting on its dispersion. DT is dispersion time and t_{wash} is the time of the wash cycle.

The dispersion time, DT, is defined by the equation:

$$DT = {}^{\tau \text{disperse}} \Big/ t_{wash}$$

where t_{wash} is the time of the wash cycle and $\tau_{disperse}$ is a time constant that is characteristic of the exponential decay of the bulk mass of powder. In essence, $\tau_{disperse}$ is the time at which ~63% of the net amount of dispersible powder will be dispersed, i.e. bulk powder added minus insoluble residues, has dispersed. The dispersion time is a dimensionless value which measures the relative dispersion over various wash cycles which vary considerably across load types and regions.

The stretching exponent, m, is a value to describe how the powder acts at its initial point of wetting, i.e., is it a slow or fast dispersing powder. The lower the value, the faster the powder initially disperses. R* is a value that describes how well the powder disperses completely. The lower the value of R* the larger the amount of the powder that is dispersed in a reasonable amount of time, such as one hour. Meanwhile, $\tau_{disperse}$ is a value that describes the kinetics of dispersion.. The lower the value of $\tau_{disperse}$ the faster the detergent disperses. Thus, it takes all three values to accurately describe the dispersion of a detergent powder. A powder may initially disperse very quickly and have a low m value but have a high residue mass (R*) because of lump-gel formation. Conversely, the powder may disperse completely but only at a slow rate, i.e. a high $\tau_{disperse}$. Accordingly, it is the inter-relation of all three variables that are needed to accurately describe the dispersability of a granular detergent.

A powdered detergent composition may be fit against this equation by measuring the amount of residual detergent, R, as a function of time, i.e. at multiple times, over the course of a wash cycle and curve fitting these values to generate a value for R*, m and $\tau_{disperse}$. The curve fitting may be performed via the use of various commercially available curve fitting software, such as for example, Excel Solver® with the constraint that R* must be some value greater than or equal to 0. Under stressed cold-water conditions defined below, a superior performing detergent composition according to the present invention will have values for the long time

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residue, R*, of less than about 20%, more preferably less about 15% and most preferably less than about 5%, values of the dispersion time, DT, of less than about 0.5, preferably less than about 0.25 and most preferably less than about 0.12 and values of the stretching exponent, m, of less than about 2, more preferably less than about 1.5 and most preferably less than about 1. Meanwhile, values of R are preferably less than 25%, more preferably less than 18% and most preferably less than about 10% by weight of the composition.

For the purposes of the present invention, the test employed to measure this residual detergent is the wire basket test, WBT which conforms to United States Pharmacopoeia Test 711 . The WBT consists of a 1 litre vessel of stainless steel, plastic, or (preferably) borosilicate glass having a stainless steel stirrer approximately 4 mm thick. A 20 mesh stationary basket (appprox. 4mm X 25mm) is made of stainless steel, being supported atop the one liter vessel by a plexiglas. One liter of distilled water at about 4.44C (40F) is placed in the dissolution vessel and circulated continuously through the vessel. A volume of granular detergent material is weighed into the stationary wire basket. The basket is then suspended midway between the agitator shaft and side wall of the apparatus. The agitator is then ran at 200 rpm. The basket is then removed from the apparatus at periodic times, with the residue transferred to a weighing dish and dried to a constant weight.

Rate of Dissolution

The rate of dissolution, ROD, according to the present invention is a measure of the dissolution rate of the surfactant chemistry itself as it is delivered in the granular detergent particles. The rate of dissolution is gauged as a percentage of total surfactant dissolved, as measured by chemical assay of the surfactant from filtered washwater. The key is to dissolve well dispersed particles quickly relative to the length of the wash cycle. Thus, the ROD predicts how quickly a detergent composition delivers its surfactant to the wash.

The ROD can be modeled using the expression:

$$U = U^* + (1-U^*)\exp\left(-\left(\frac{t}{RT(t_{wash})}\right)^n\right)$$

where U is the fraction of undissolved surfactant at any point in time, t, U* is the long term surfactant residual, i.e. insoluble surfactant particles and/or precipitates, t is any single point in

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time, n is a stretching exponent that describes how the surfactant acts, i.e. dissolves, at the early stages of wetting in the test. RT is dissolution time and t_{wash} is the time of the wash cycle.

The dissolution time, RT, is defined by the equation:

$$RT = \tau ROD / t_{wash}$$

where t_{wash} is the time of the wash cycle and τ_{ROD} is a time constant that is characteristic of the dissolution of surfactant. In essence, τ_{ROD} is the time at which ~63% of the net amount of soluble surfactant will dissolve, i.e. total surfactant added minus insoluble surfactant residues, has dissolved. The dissolution time is a dimensionless value which measures the relative dissolution over various wash cycles which vary considerably across load types and regions. Thus, RT provides for an accurate prediction of the dissolution of a powdered detergent in differing wash conditions and cycles.

The stretching exponent, m, is a value to describe how the surfactant acts at its initial point of wetting, i.e., is it a slow or fast wetting surfactant. The lower the value, the faster the surfactant initially dissolves. U* is a value that describes how well the surfactant dissolves completely. The lower the value of U* the larger the amount of the surfactant that is soluble in a reasonable amount of time, such as one hour. Meanwhile, τ_{ROD} is a value that describes how long the surfactant takes to ultimately dissolve. The lower the value of τ_{ROD} the faster the surfactant dissolves. Thus, as in dispersion, it takes all three values to accurately describe the dissolution of surfactant particles in a wash solution.

A powdered detergent composition may be fit against this equation by measuring the amount of residual surfactant, U, as a function of time, i.e. at multiple times, over the course of a wash cycle and curve fitting these values to generate a value for U*, n and τ_{ROD}. The curve fitting may be performed as in the dispersion calculations via the use of various commercially available curve fitting software, such as for example, Excel Solver® with the constraint that U* must be some value greater than or equal to 0. Under stressed cold-water conditions defined below, a superior performing detergent composition according to the present invention will have values for the long time residue, U*, of less than about 14%, more preferably less about 7% and most preferably less than about 3.5%, values of the dispersion time, RT, of less than about 0.5, preferably less than about 0.25 and most preferably less than about 0.12 and values of the stretching exponent, n, of less than about 2, more preferably less than about 1.5 and most preferably less than about 1. Meanwhile values for U are typically greater than 30% in 2

minutes and 70 % in 5 minutes, more preferably greater than 40% in 2 minutes and 80% in 5 minutes and most preferably greater than 50% in 2 minutes and 90% in 5 minutes.

For the purposes of the present invention, the test employed to measure the residual surfactant is described by the following procedure which conforms to United States Pharmacopoeia test 711. A 1 liter vessel of stainless steel, plastic, or (preferably) borosilicate glass having a stainless steel stirrer approximately 4 mm thick is provided. One liter of distilled water at about 10°C (50°F) is placed in the dissolution vessel and circulated continuously through the vessel. A 10.0 gram (+/- 0.1 gram) sample is decanted into the water with the agitator turning at 200 rpm. At specific time intervals (30 seconds, 1, 2.5 and 5 minutes), a 10 cc sample is extracted and immediately filtered through 0.45 micron filter paper. After filtering the last sample, increase the agitator speed to 300 rpm for the remainder of the test. Two 10 cc samples are extracted 5 minutes later. One sample is filtered, the other sample is not filtered. This unfiltered sample is the "100% dissolved". This unfiltered sample will be used to generate the "100% dissolved" control. Place this sample into a 60°C (140°F) oven for at least one hour to ensure full dissolution of surfactant. Remove sample from oven, agitate to ensure homogeneity, and then filter with a clean syringe and filter into a clean vial. This is now the "100% dissolved" control. Measure the surfactant level in each sample by conventional means such as for example titration to determine cationic SO_3 in sulfate containing anionic surfactants. Convert the raw results into "% dissolved" by dividing the results by the "100% dissolved" result and multiplying by 100.

Insoluble Residues

Insoluble Residues is a measure of the amount of particles in the granular detergent which will not dissolve in a reasonable period of time such as the length of the wash cycle. Insoluble residues larger than a certain size will wind up as visible specks or particles on fabrics as they are filtered from the wash water by the fabrics. The insoluble residues can be measured in the washwater before they are filtered through the fabrics. The measurements may be performed by conventional light scattering devices that use Fraunhoffer light scattering principles, such as for example Malvern or Horiba particle size analyzers. The unfiltered washwater is passed through a diffraction cell where dispersed particles scatter light passed through the cell. The degree of scattering is proportional to the size of the dispersed particles. The spectrum of scattering allows the measurement of the full particle size distribution of the residues in the wash water. A superior performing detergent composition will have a D90 of insoluble residues of less than about 15μm and more preferably a D90 of less than about 10μm.

That is at least 90% of the insoluble residues in the washwater have a particle size of less than about 15μm and more preferably less than about 10μm.

Detergent Compositions

Detergent compositions which satisfy the transition profiles as disclosed above may be formulated with a varying array of ingredients and properties to achieve the overall superior solubility profile as defined by the transitions discussed above.

The granular detergent composition may achieve the desired benefits of solubility, improved aesthetics and flowability via optimal selection of the geometric mean particle diameter of certain levels of particles in the composition. By "improved aesthetics", it is meant that the consumer views a granular detergent product which has a more uniform appearance of particles as opposed to past granular detergent products which contained particles of varying size and composition. To that end, at least about 50%, more preferably at least about 75%, even more preferably at least about 90%, and most preferably at least about 95%, by weight of the total particles in the detergent product, have the selected mean particle size diameter. In this way, a substantial portion of the granular detergent product will have the uniform size so as to provide the aesthetic appearance desired by consumers.

Preferably, the geometric mean particle diameter of the particles is from about 500 microns to about 1500 microns, more preferably from about 600 microns to about 1200 microns, and most preferably from about 700 microns to about 1000 microns. The particle size distribution is defined by a relative tight geometric standard deviation or "span" so as not to have too many particles outside of the target size. Accordingly, the geometric standard deviation is preferably is from about 1 to about 2, more preferably is from about 1.0 to about 1.7, even more preferably is from about 1.0 to about 1.4, and most preferably is from about 1.0 to about 1.2. The average bulk density of the particles is preferably at least about 450 g/l, more preferably at least about 550 g/l, and most preferably at least about 650 g/l.

While not intending to be bound by theory, it is believed that solubility is enhanced as a result of the particles in the detergent composition being more of the same size. Specifically, as a result of the particles being more uniform in size, the actual "contact points" among the particles in the detergent composition is reduced which, in turn, reduces the "bridging effect" commonly associated with the "lump-gel" dissolution difficulties of granular detergent compositions. Previous granular detergent compositions contained particles of varying sizes which leads to more contact points among the particles. For example, a large particle could

have many smaller particles in contact with it rendering the particle site ripe for lump-gel formation. The level and uniform size of the particles in the granular detergent composition of the present invention avoids such problems.

By "a portion" of the particles, it is meant that at least some particles in the detergent composition contain a detersive surfactant and/or a detergent builder to provide the fundamental building blocks of a typical detergent composition. The various surfactants and builders as well as their respective levels in the composition are set forth hereinafter. Typically, the detergent composition will contain from about 1% to about 50% by weight of a detersive surfactant and from about 1% to about 75% by weight of a detergent builder.

Alternatively, the transition profile of the present invention may be satisfied via a more standard or uniform shape of the individual particles of the granular detergent. A more uniform shape leads to more uniform dispersion and a reduction in the contact points between particles as discussed above. Shape can be measured in a number of different ways known to those of ordinary skill in the art. One such method is using optical microscopy with Optimus (V5.0) image analysis software. Important calculated parameters are:

"Circularity" which is defined as (measured perimeter length of the particle image)^2/(measured area of the particle image). The circularity of a perfectly smooth sphere (minimum circularity) is 12.57; and

"Aspect Ratio" which is defined as the length/width of the particle image.

Each of these attributes is important and can be averaged over the bulk granular detergent composition. Further, the combination of the two parameters as defined by the product of the parameters is important as well (i.e. both must be controlled to get a product with good appearance).

Preferably, the granular detergent compositions of this invention have circularity less than about 50, preferably less than about 30, more preferably less than about 23, most preferably less than about 18. Also preferred are granular detergent compositions with aspect ratios less than about 2, preferably less than about 1.5, more preferably less than about 1.3 most preferably less than about 1.2.

Additionally, it is preferred to have a uniform distribution of shapes among the particles in the composition. Specifically, the granular detergent compositions of this invention have a standard deviation of the number distribution of circularity less than about 20, that is preferably less than about 10, more preferably less than about 7 most preferably less than about 4. And the standard deviation of the number distribution of aspect ratios is preferably less than about 1,

more preferably less than about 0.5, even more preferably less than about 0.3, most preferably less than about 0.2.

In an especially preferred process of the present invention, granular detergent compositions are produced wherein the product of circularity and aspect ratio is less than about 100, preferably less than about 50, more preferably less than about 30, and most preferably less than about 20. Also preferred are granular detergent compositions with the standard deviation of the number distribution of the product of circularity and aspect ratio of less than about 45, preferably less than about 20, more preferably less than about 7 most preferably less than about 2.

Yet another detergent design method for achieving granular detergents satisfying the aforementioned transition profile involves the use of homogeneous detergents compositions wherein the homogeneous detergent contributes to the aforementioned benefits. The homogeneity number describes the distribution of ingredients within a specific particles and between particles in a composition. In the past, it was believed that homogeneous distribution of key ingredients such as surfactant both within the particle and between particles was optimal. Thus, detergent composition would consist of a uniform particle of the same ingredients, such as spray-dried detergent ingredients and have significant solubility drawbacks. In recent years, detergent compositions have consisted of differing particles of dual particle systems. However, these particles where differ composition and form, e.g. spray-dried granules and agglomerates. These detergent products also experience solubility drawbacks.

However, a detergent composition that has a homogeneity number of less than about 0.5 or greater than about 1.0, more preferably, greater than 1.25 and most preferably greater than about 1.5 have superior solubility profiles per the present invention. The homogeneity number is represented by the formula:

$$HN = X_{bulk}/X_{part}$$

where X_{bulk} measure of the homogeneity of the particles in the compositions while X_{part} is the measure of the homogeneity of the individual particles. Thus, X_{bulk} is the ratio of the concentration of the selected ingredient in the particle with the lowest levels of that ingredient to the concentration of the selected ingredient in the particle with the highest levels of the selected ingredient and X_{part} is the ratio of the concentration in the discrete area with the lowest amount of the selected ingredient to the concentration in the discrete area of the particle having the

highest amounts of the selected ingredient, of less than about 0.5 or greater than about 1, preferably greater than about 1.25 and more preferably greater than about 1.5.

Thus, in a detergent composition, X_{bulk} is the ratio of the concentration of a selected detergent ingredient such as surfactant, builder, etc. in the particles in the composition with the lowest levels of the selected ingredient to the concentration of the selected ingredient in the particles with the highest level of the selected ingredient. This provides the homogeneity between particles in the composition. Thus, X_{bulk} is represent by the formula:

$$X_{bulk} = X_{min}/X_{max}$$

where X_{min} is the concentration of the selected ingredient in the particles in the composition with the lowest levels of the selected ingredient and X_{max} concentration of a selected detergent ingredient in the particles in the composition with the highest levels of the selected ingredient. For example, for a detergent composition in which are the particles have the same concentration such as a spray-dried granule with a active concentration of 25% surfactant, X_{bulk} would be equal to one (1) or 0.25/0.25. However, in a composition which comprises a spray dried granule of 20% active surfactant and a detergent agglomerate of 30% detergent active X_{bulk} would be equal to 0.67 or 0.2/0.3.

X_{partk} is the ratio of the concentration of a selected detergent ingredient such as surfactant, builder, etc. in the same particle, or in other words a measure of the homogeneity of the individual particle. X_{part} is the ratio of the selected ingredient in discrete areas of the particle. X_{part} is the ratio of the concentration in the discrete with the lowest concentration of the ingredient to the concentration of the selected ingredient in the discrete area with the highest concentration within the same particle. Thus, X_{part} is represent by the formula:

$$X_{part} = X_{min}/X_{max}$$

where X_{min} is the concentration of the selected ingredient in the discrete area in the particle with the lowest levels of the selected ingredient and X_{max} concentration of a selected detergent ingredient in the discrete areas in the particle with the highest levels of the selected ingredient. A discrete area of the present invention is one in which the is a clear morphological difference between the areas and is typically an area that accounts for more than 1%, preferably, 5% the volume of the particle. For example, a particle that is homogeneous throughout the particle has only one (1) discrete are. Thus, a particles which has the same concentration throughout such as

a spray-dried granule with a active concentration of 25% surfactant, X_{part} would be equal to one (1) or 0.25/0.25 as the particle contains only one discrete area. However, in a particle, which is agglomerated from two different starting ingredients such as spray-dried granules having 5% active surfactant and dry detergent agglomerates having 50% active surfactant to form mixed agglomerates as defined herein, X_{bulk} would be equal to 0.1 or 0.05/0.5.

The homogeneity number of the present invention is to be calculated on particles which comprise the bulk of the detergent composition. Thus, particles which individually or collectively account for less than about 10% by weight of the finished composition should not be employed in the calculation of homogeneity number. The ingredient typically include admix ingredients such for example, enzymes, bleach ingredients, perfumes ingredients, and various other minor additions.

While not wishing to be bound by theory, it is believed that by concentrating certain ingredients and/or selectively separating them ,one can prevent the gelling upon dissolution due to chemical interactions between the particles. Such ingredients include separation of surfactants such as alcohol based surfactants and alkyl benzene sulphonates from one another and/or grouping electrolytes with one type of surfactant leads to improved solubility. By way of example, a fully formulated detergent composition which comprises a surfactant system having an electrolyte rich surfactant zone and an electrolyte poor surfactant zone may be employed to provide a granular detergent with improved solubility. The composition may be in the form of a single particle with separate discrete surfactant zones or may be in the form of multiple particles wherein each surfactant zone is represented by a separate particle. The electrolyte poor surfactant zone comprises less than about 20%, more preferably less than about 10%, even more preferably less than about 2% and most preferably about 0% electrolyte in conjunction with a surfactant or blend of surfactants selected from the class of alcohol sulfate surfactants. Meanwhile, the electrolyte rich surfactant zone comprises more than about 20%, more preferably more than about 35% and most preferably more than about 45% electrolyte in conjunction with a surfactant or blend of surfactants selected from the class of alkyl benzene sulfonate surfactants. Thus, via zone separation wherein the electrolyte is separated from the proximity of the alcohol sulfate surfactants, the formation of lump-gel residues is minimized and/or reduced resulting in both superior dissolution and dispersion profiles for the granular detergent of the present invention.

Yet another method of improving solubility of granular detergent compositions is to selectively coat with a coating agent the particles and in particular particles that include

ingredients that are sticky in nature such as surfactants. Such coating methods are well known to those of ordinary skill in the art, for example spray drums.

Lastly, the solubility of a granular detergent composition may be impacted by the methods employed to manufacture the granular powder. Granular compositions are typically designed to provide an in the wash pH of from about 7.5 to about 11.5, more preferably from about 9.5 to about 10.5. Low density compositions can be prepared by standard spray-drying processes. Various means and equipment are available to prepare high density compositions. Current commercial practice in the field employs spray-drying towers to manufacture compositions which have a density less than about 500 g/l. Accordingly, if spray-drying is used as part of the overall process, the resulting spray-dried particles must be further densified using the means and equipment described hereinafter. In the alternative, the formulator can eliminate spray-drying by using mixing, densifying and granulating equipment that is commercially available. The following is a nonlimiting description of such equipment suitable for use herein.

Various means and equipment are available to prepare high density (i.e., greater than about 500, preferably greater than about 600, grams/liter or "g/l"), high solubility, free-flowing, granular detergent compositions according to the present invention. Current commercial practice in the field employs spray-drying towers to manufacture granular laundry detergents which often have a density less than about 500 g/l. In this procedure, an aqueous slurry of various heat-stable ingredients in the final detergent composition are formed into homogeneous granules by passage through a spray-drying tower, using conventional techniques, at temperatures of about 175°C to about 225°C. However, if spray drying is used as part of the overall process herein, additional process steps as described hereinafter must be used to obtain the level of density (i.e., > 600 g/l) required by modern compact, low dosage detergent products.

For example, spray-dried granules from a tower can be densified further by loading a liquid such as water or a nonionic surfactant into the pores of the granules and/or subjecting them to one or more high speed mixer/densifiers. A suitable high speed mixer/densifier for this process is a device marketed under the tradename "Lödige CB 30" or "Lödige CB 30 Recycler" which comprises a static cylindrical mixing drum having a central rotating shaft with mixing/cutting blades mounted thereon. In use, the ingredients for the detergent composition are introduced into the drum and the shaft/blade assembly is rotated at speeds in the range of 100-2500 rpm to provide thorough mixing/densification. See Jacobs et al, U.S. Patent 5,149,455, issued September 22, 1992. The preferred residence time in the high speed

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mixer/densifier is from about 1 to 60 seconds. Other such apparatus includes the devices marketed under the tradename "Shugi Granulator" and under the tradename "Drais K-TTP 80).

Another process step which can be used to densify further spray-dried granules involves grinding and agglomerating or deforming the spray-dried granules in a moderate speed mixer/densifier so as to obtain particles having lower intraparticle porosity. Equipment such as that marketed under the tradename "Lödige KM" (Series 300 or 600) or "Lödige Ploughshare" mixer/densifiers are suitable for this process step. Such equipment is typically operated at 40-160 rpm. The residence time of the detergent ingredients in the moderate speed mixer/densifier is from about 0.1 to 12 minutes. Other useful equipment includes the device which is available under the tradename "Drais K-T 160". This process step which employs a moderate speed mixer/densifier (e.g. Lödige KM) can be used by itself or sequentially with the aforementioned high speed mixer/densifier (e.g. Lödige CB) to achieve the desired density. Other types of granules manufacturing apparatus useful herein include the apparatus disclosed in U.S. Patent 2,306,898, to G. L. Heller, December 29, 1942.

While it may be more suitable to use the high speed mixer/densifier followed by the low speed mixer/densifier, the reverse sequential mixer/densifier configuration is also contemplated by the invention. One or a combination of various parameters including residence times in the mixer/densifiers, operating temperatures of the equipment, temperature and/or composition of the granules, the use of adjunct ingredients such as liquid binders and flow aids, can be used to optimize densification of the spray-dried granules in the process of the invention. By way of example, see the processes in Appel et al, U.S. Patent 5,133,924, issued July 28, 1992 (granules are brought into a deformable state prior to densification); Delwel et al, U.S. Patent 4,637,891, issued January 20, 1987 (granulating spray-dried granules with a liquid binder and aluminosilicate); Kruse et al, U.S. Patent 4,726,908, issued February 23, 1988 (granulating spray-dried granules with a liquid binder and aluminosilicate); and, Bortolotti et al, U.S. Patent 5,160,657, issued November 3, 1992 (coating densified granules with a liquid binder and aluminosilicate).

In those situations in which particularly heat sensitive or highly volatile detergent ingredients are to be incorporated into the final detergent composition, processes which do not include spray drying towers are preferred. The formulator can eliminate the spray-drying step by feeding, in either a continuous or batch mode, starting detergent ingredients directly into mixing/densifying equipment that is commercially available. One particularly preferred embodiment involves charging a surfactant paste and an anhydrous builder material into a high

speed mixer/densifier (e.g. Lödige CB) followed by a moderate speed mixer/densifier (e.g. Lödige KM) to form high density detergent agglomerates. See Capeci et al, U.S. Patent 5,366,652, issued November 22, 1994 and Capeci et al, U.S. Patent 5,486,303, issued January 23, 1996. Optionally, the liquid/solids ratio of the starting detergent ingredients in such a process can be selected to obtain high density agglomerates that are more free flowing and crisp.

Optionally, the process may include one or more recycle streams of undersized particles produced by the process which are fed back to the mixer/densifiers for further agglomeration or build-up. The oversized particles produced by this process can be sent to grinding apparatus and then fed back to the mixing/densifying equipment. These additional recycle process steps facilitate build-up agglomeration of the starting detergent ingredients resulting in a finished composition having a uniform distribution of the desired particle size (400-700 microns) and density (> 550 g/l). See Capeci et al, U.S. Patent 5,516,448, issued May 14, 1996 and Capeci et al, U.S. Patent 5,489,392, issued February 6, 1996. Other suitable processes which do not call for the use of spray-drying towers are described by Bollier et al, U.S. Patent 4,828,721, issued May 9, 1989; Beerse et al, U.S. Patent 5,108,646, issued April 28, 1992; and, Jolicoeur, U.S. Patent 5,178,798, issued January 12, 1993.

In yet another embodiment, the high density detergent composition of the invention can be produced using a fluidized bed mixer. In this process, the various ingredients of the finished composition are combined in an aqueous slurry (typically 80% solids content) and sprayed into a fluidized bed to provide the finished detergent granules. Prior to the fluidized bed, this process can optionally include the step of mixing the slurry using the aforementioned Lödige CB mixer/densifier or a "Flexomix 160" mixer/densifier, available from Shugi. Fluidized bed or moving beds of the type available under the tradename "Escher Wyss" can be used in such processes.

Another suitable process which can be used herein involves feeding a liquid acid precursor of an anionic surfactant, an alkaline inorganic material (e.g. sodium carbonate) and optionally other detergent ingredients into a high speed mixer/densifier (residence time 5-30 seconds) so as to form agglomerates containing a partially or totally neutralized anionic surfactant salt and the other starting detergent ingredients. Optionally, the contents in the high speed mixer/densifier can be sent to a moderate speed mixer/densifier (e.g. Lödige KM) for further agglomeration resulting in the finished high density detergent composition. See Appel et al, U.S. Patent 5,164,108, issued November 17, 1992.

Optionally, high density detergent compositions according to the invention can be produced by blending conventional or densified spray-dried detergent granules with detergent agglomerates in various proportions (e.g. a 60:40 weight ratio of granules to agglomerates) produced by one or a combination of the processes discussed herein. Additional adjunct ingredients such as enzymes, perfumes, brighteners and the like can be sprayed or admixed with the agglomerates, granules or mixtures thereof produced by the processes discussed herein. Bleaching compositions in granular form typically limit water content, for example, to less than about 7% free water, for best storage stability.

In yet another optional processing step, starting ingredients comprising spray-dried granules and agglomerates formed as described above may be combined, optionally with other starting ingredients, such as carbonate, etc, and liquid binder material in a high, moderate or low speed mixer and agglomerated into a single mixed agglomerate particle to provide a detergent with a uniform size and distribution. The mixed agglomerate may be formed in a single mixer or series of mixers including moderate speed mixer/densifiers in combination with fluid bed granulators.

Of course, one of ordinary skill in the art will recognize that the above mentioned detergent formulation methods are merely a representative example of the techniques that may be employed to design a superior performing and highly soluble granular detergent and that any one or a combination of these techniques and/or other methods may be employed.

DETERGENT COMPONENTS

The surfactant system of the detergent composition may include anionic, nonionic, zwitterionic, ampholytic and cationic classes and compatible mixtures thereof. Detergent surfactants are described in U.S. Patent 3,664,961, Norris, issued May 23, 1972, and in U.S. Patent 3,919,678, Laughlin et al., issued December 30, 1975, both of which are incorporated herein by reference. Cationic surfactants include those described in U.S. Patent 4,222,905, Cockrell, issued September 16, 1980, and in U.S. Patent 4,239,659, Murphy, issued December 16, 1980, both of which are also incorporated herein by reference.

Nonlimiting examples of surfactant systems include the conventional C_{11}-C_{18} alkyl benzene sulfonates ("LAS") and primary, branched-chain and random C_{10}-C_{20} alkyl sulfates ("AS"), the C_{10}-C_{18} secondary (2,3) alkyl sulfates of the formula $CH_3(CH_2)_x(CHOSO_3^-M^+)$ CH_3 and $CH_3(CH_2)_y(CHOSO_3^-M^+)CH_2CH_3$ where x and (y + 1) are integers of at least about 7, preferably at least about 9, and M is a water-solubilizing cation, especially sodium, unsaturated sulfates such as oleyl sulfate, the C_{10}-C_{18} alkyl alkoxy sulfates ("AE_xS"; especially

EO 1-7 ethoxy sulfates), C_{10}-C_{18} alkyl alkoxy carboxylates (especially the EO 1-5 ethoxycarboxylates), the $C_{10\text{-}18}$ glycerol ethers, the C_{10}-C_{18} alkyl polyglycosides and their corresponding sulfated polyglycosides, and C_{12}-C_{18} alpha-sulfonated fatty acid esters. If desired, the conventional nonionic and amphoteric surfactants such as the C_{12}-C_{18} alkyl ethoxylates ("AE") including the so-called narrow peaked alkyl ethoxylates and C_6-C_{12} alkyl phenol alkoxylates (especially ethoxylates and mixed ethoxy/propoxy), C_{12}-C_{18} betaines and sulfobetaines ("sultaines"), C_{10}-C_{18} amine oxides, and the like, can also be included in the surfactant system. The C_{10}-C_{18} N-alkyl polyhydroxy fatty acid amides can also be used. Typical examples include the C_{12}-C_{18} N-methylglucamides. See WO 9,206,154. Other sugar-derived surfactants include the N-alkoxy polyhydroxy fatty acid amides, such as C_{10}-C_{18} N-(3-methoxypropyl) glucamide. The N-propyl through N-hexyl C_{12}-C_{18} glucamides can be used for low sudsing. C_{10}-C_{20} conventional soaps may also be used. If high sudsing is desired, the branched-chain C_{10}-C_{16} soaps may be used. Mixtures of anionic and nonionic surfactants are especially useful. Other conventional useful surfactants are listed in standard texts.

The detergent composition can, and preferably does, include a detergent builder. Builders are generally selected from the various water-soluble, alkali metal, ammonium or substituted ammonium phosphates, polyphosphates, phosphonates, polyphosphonates, carbonates, silicates, borates, polyhydroxy sulfonates, polyacetates, carboxylates, and polycarboxylates. Preferred are the alkali metal, especially sodium, salts of the above. Preferred for use herein are the phosphates, carbonates, silicates, $C_{10\text{-}18}$ fatty acids, polycarboxylates, and mixtures thereof. More preferred are sodium tripolyphosphate, tetrasodium pyrophosphate, citrate, tartrate mono- and di-succinates, sodium silicate, and mixtures thereof (see below).

Specific examples of inorganic phosphate builders are sodium and potassium tripolyphosphate, pyrophosphate, polymeric metaphosphate having a degree of polymerization of from about 6 to 21, and orthophosphates. Examples of polyphosphonate builders are the sodium and potassium salts of ethylene diphosphonic acid, the sodium and potassium salts of ethane 1-hydroxy-1, 1-diphosphonic acid and the sodium and potassium salts of ethane, 1,1,2-triphosphonic acid. Other phosphorus builder compounds are disclosed in U.S. Patents 3,159,581; 3,213,030; 3,422,021; 3,422,137; 3,400,176 and 3,400,148, all of which are incorporated herein by reference.

Examples of nonphosphorus, inorganic builders are sodium and potassium carbonate, bicarbonate, sesquicarbonate, tetraborate decahydrate, and silicates having a weight ratio of SiO_2 to alkali metal oxide of from about 0.5 to about 4.0, preferably from about 1.0 to about 2.4. Water-soluble, nonphosphorus organic builders useful herein include the various alkali metal, ammonium and substituted ammonium polyacetates, carboxylates, polycarboxylates and polyhydroxy sulfonates. Examples of polyacetate and polycarboxylate builders are the sodium, potassium, lithium, ammonium and substituted ammonium salts of ethylene diamine tetraacetic acid, nitrilotriacetic acid, oxydisuccinic acid, mellitic acid, benzene polycarboxylic acids, and citric acid.

Polymeric polycarboxylate builders are set forth in U.S. Patent 3,308,067, Diehl, issued March 7, 1967, the disclosure of which is incorporated herein by reference. Such materials include the water-soluble salts of homo- and copolymers of aliphatic carboxylic acids such as maleic acid, itaconic acid, mesaconic acid, fumaric acid, aconitic acid, citraconic acid and methylenemalonic acid. Some of these materials are useful as the water-soluble anionic polymer as hereinafter described, but only if in intimate admixture with the nonsoap anionic surfactant.

Other suitable polycarboxylates for use herein are the polyacetal carboxylates described in U.S. Patent 4,144,226, issued March 13, 1979 to Crutchfield et al., and U.S. Patent 4,246,495, issued March 27, 1979 to Crutchfield et al., both of which are incorporated herein by reference. These polyacetal carboxylates can be prepared by bringing together under polymerization conditions an ester of glyoxylic acid and a polymerization initiator. The resulting polyacetal carboxylate ester is then attached to chemically stable end groups to stabilize the polyacetal carboxylate against rapid depolymerization in alkaline solution, converted to the corresponding salt, and added to a detergent composition. Particularly preferred polycarboxylate builders are the ether carboxylate builder compositions comprising a combination of tartrate monosuccinate and tartrate disuccinate described in U.S. Patent 4,663,071, Bush et al., issued May 5, 1987, the disclosure of which is incorporated herein by reference.

Water-soluble silicate solids represented by the formula $SiO_2 \bullet M_2O$, M being an alkali metal, and having a $SiO_2:M_2O$ weight ratio of from about 0.5 to about 4.0, are useful salts in the detergent granules of the invention at levels of from about 2% to about 15% on an anhydrous weight basis, preferably from about 3% to about 8%. Anhydrous or hydrated particulate silicate can be utilized, as well.

Any number of additional ingredients can also be included as components in the granular detergent composition. These include other detergency builders, bleaches, bleach activators, suds boosters or suds suppressors, anti-tarnish and anti-corrosion agents, soil suspending agents, soil release agents, germicides, pH adjusting agents, nonbuilder alkalinity sources, chelating agents, smectite clays, enzymes, enzyme-stabilizing agents and perfumes. See U.S. Patent 3,936,537, issued February 3, 1976 to Baskerville, Jr. et al., incorporated herein by reference.

Bleaching agents and activators are described in U.S. Patent 4,412,934, Chung et al., issued November 1, 1983, and in U.S. Patent 4,483,781, Hartman, issued November 20, 1984, both of which are incorporated herein by reference. Chelating agents are also described in U.S. Patent 4,663,071, Bush et al., from Column 17, line 54 through Column 18, line 68, incorporated herein by reference. Suds modifiers are also optional ingredients and are described in U.S. Patents 3,933,672, issued January 20, 1976 to Bartoletta et al., and 4,136,045, issued January 23, 1979 to Gault et al., both incorporated herein by reference.

Suitable smectite clays for use herein are described in U.S. Patent 4,762,645, Tucker et al., issued August 9, 1988, Column 6, line 3 through Column 7, line 24, incorporated herein by reference. Suitable additional detergency builders for use herein are enumerated in the Baskerville patent, Column 13, line 54 through Column 16, line 16, and in U.S. Patent 4,663,071, Bush et al., issued May 5, 1987, both incorporated herein by reference.

The following examples are presented for illustrative purposes only and are not to be construed as limiting the scope of the appended claims in any way.

Abbreviations used in Examples

In the detergent compositions, the abbreviated component identifications have the following meanings:

LAS	:	Sodium linear C11-13 alkyl benzene sulfonate
TAS	:	Sodium tallow alkyl sulfate
C45AS	:	Sodium C14 - C15 alkyl sulfate
C45E3S	:	Sodium C14-C15 alkyl sulfate condensed with 3 moles of ethylene oxide
QAS	:	$R2.N+(CH3)2(C2H4OH)$ with $R2 = C12 - C14$

Zeolite A	:	Hydrated sodium aluminosilicate of formula Na12(A1O2SiO2)12.27H2O having a primary particle size in the range from 0.1 to 10 micrometers (weight expressed on an anhydrous basis)
NaSKS-6	:	Crystalline layered silicate of formula δ- Na2Si2O5
Citric acid	:	Anhydrous citric acid
Carbonate	:	Anydrous sodium carbonate with a particle size between 200μm and 900μm
Bicarbonate	:	Anhydrous sodium bicarbonate with a particle size distribution between 400μm and 1200μm
Sulfate	:	Anhydrous sodium sulfate
Mg sulfate	:	Anhydrous magnesium sulfate
Citrate	:	Tri-sodium citrate dihydrate of activity 86.4% with a particle size distribution between 425μm and 850μm
MA/AA	:	Copolymer of 1:4 maleic/acrylic acid, average molecular weight about 70,000
AA	:	Sodium polyacrylate polymer of average molecular weight 4,500
Protease	:	Proteolytic enzyme, having 4% by weight of active enzyme, as described in WO 95/10591, sold by Genencor Int. Inc.
Cellulase	:	Cellulytic enzyme, having 0.23% by weight of active enzyme, sold by NOVO Industries A/S under the tradename Carezyme
Amylase	:	Amylolytic enzyme, having 1.6% by weight of active enzyme, sold by NOVO Industries A/S under the tradename Termamyl 120T
Lipase	:	Lipolytic enzyme, having 2.0% by weight of active enzyme, sold by NOVO Industries A/S under the tradename Lipolase
Perborate	:	Sodium perborate
Percarbonate	:	Sodium percarbonate
NOBS	:	Nonanoyloxybenzene sulfonate in the form of the sodium salt
NAC-OBS	:	(6-nonamidocaproyl) oxybenzene sulfonate
TAED	:	Tetraacetylethylenediamine
DTPA	:	Diethylene triamine pentaacetic acid
EDDS	:	Ethylenediamine-N,N'-disuccinic acid, (S,S) isomer in the form of its sodium salt.

Photoactivated	:	Sulfonated zinc phthlocyanine encapsulated in bleach (1) dextrin soluble polymer
Brightener	:	Disodium 4,4'-bis(4-anilino-6-morpholino-1.3.5-triazin-2-yl)amino) stilbene-2:2'-disulfonate
HEDP	:	1,1-hydroxyethane diphosphonic acid
PEGx	:	Polyethylene glycol, with a molecular weight of x (typically 4,000)
QEA	:	bis((C2H5O)(C2H4O)n)(CH3) -N+-C6H12-N+-(CH3) bis((C2H5O)-(C2H4 O))n, wherein n = from 20 to 30
SRP 2	:	Diethoxylated poly (1, 2 propylene terephtalate) short block polymer
Silicone antifoam		Polydimethylsiloxane foam controller with siloxane-oxyalkylene copolymer as dispersing agent with a ratio of said foam controller to said dispersing agent of 10:1 to 100:1

In the following examples all levels are quoted as % by weight of the composition:

Example I

The following compositions are in accordance with the invention.

	A	B	C	D	E	F	G	H	I
Spray-dried Granules									
LAS	10.0	10.0	15.0	5.0	5.0	10.0	-	-	3.1
TAS	-	1.0	-				-	-	-
$C_{45}AS$	-	-	1.0		2.0	2.0	-	-	-
$C_{45}AE_3S$	-	-		1.0			-	-	-
QAS			1.0	1.0			-	-	-
DTPA, HEDP and/or EDDS	0.3	0.3	0.5	0.3			-	-	0.7
MgSO4	0.5	0.5	0.1	-			-	-	-
Sodium citrate	-	-	-	3.0	5.0		-	-	-
Sodium carbonate	10.0	7.0	15.0			10.0	-	-	10.4
Sodium sulphate	5.0	5.0	-	-	5.0	3.0	-	-	1.2
Sodium silicate 1.6R	-	-	-	-	2.0		-	-	-
Zeolite A	16.0	18.0	20.0	20.0	-	-	-	-	22.9

SKS-6	-	-	-	3.0	5.0	-	-	-	-
MA/AA or AA	1.0	2.0	11.0	-	-	2.0	-	-	-
PEG 4000	-	2.0	-	1.0	-	1.0	-	-	0.5
QEA	1.0	-	-	-	1.0	-	-	-	-
Brightener	0.05	0.05	0.05	-	0.05	-	-	-	0.11
Silicone oil	0.01	0.01	0.01	-	-	0.01	-	-	0.02
Agglomerate									
LAS			-	-	-	-	2.0	2.0	1.0
C$_{45}$AS			-	-	-	-	2.0	-	11.0
AE$_3$			-	-	-	-	-	1.0	-
Carbonate			-	-	4.0	1.0	1.0	1.0	7.7
Citric acid			-	-	-	4.0	-	1.0	-
QEA			-	-	-	2.0	2.0	1.0	-
SRP			-	-	-	1.0	1.0	0.2	-
Zeolite A			-	-	-	15.0	26.0	15.0	14.1
PEG	-	-	-	-	-	-	4.0	-	1.2
Builder Agglomerates									
SKS-6	6.0	-	-	-	6.0	3.0	-	7.0	-
LAS	4.0	5.0	-	-	5.0	3.0	-	10.0	-
Dry-add particulate components									
Malic acid/carbonate/bicarbonate (40:20:40)	8.0	10.0	10.0	4.0	-	8.0	2.0	2.0	10.0
QEA	-	-	-	0.2	0.5	-	-	-	-
NACAOBS	3.0	-	-	1.5	-	-	-	2.5	-
NOBS	-	3.0	3.0	-	-	-	-	-	2.4
TAED	2.5	-	-	1.5	2.5	6.5	-	1.5	-

LAS (flake)	10.0	10.0	-	-	-	-	-	8.0	-
Spray-on									
Brightener	0.2	0.2	0.3	0.1	0.2	0.1	-	0.6	-
Dye	-	-	-	0.3	0.05	0.1	-	-	-
AE7	-	-	-	-	-	0.5	-	0.7	-
Perfume	-	-	-	0.8	-	0.5	-	0.5	-
Dry-add									
Citrate	-	-	20.0	4.0	-	5.0	15.0	-	-
Percarbonate	15.0	3.0	6.0	10.0	-	-	-	18.0	2.5
Perborate	-	-	-	-	6.0	18.0	-	-	-
Photobleach	0.02	0.02	0.02	0.1	0.05	-	0.3	-	-
Enzymes (cellulase, amylase, protease, lipase)	1.3	0.3	0.5	0.5	0.8	2.0	0.5	0.16	0.13
Carbonate	0.0	10.0	-	-	-	5.0	8.0	10.0	-
Perfume (encapsulated)	0.6	0.5	0.5	-	0.3	0.5	0.2	0.1	0.4
Suds suppressor	1.0	0.6	0.3	-	0.10	0.5	1.0	0.3	0.3
Soap	0.5	0.2	0.3	3.0	0.5	-	-	0.3	-
Citric acid	-	-	-	6.0	6.0	-	-	-	-
Dyed carbonate (blue, green)	0.5	0.5	1.0	2.0	-	0.5	0.5	0.5	-
SKS-6	-	-	-	4.0	-	-	-	6.0	-
Fillers up to 100%									

The compositions exemplified above have at least 90% by weight of particles having a geometric mean particle diameter of from about 850 microns with a geometric standard deviation of from about 1.2. Unexpectedly, the compositions have improved aesthetics, flowability and solubility. For example, formula I has a dispersion wherein R* is less than 1%, m is 0.86, $t_{disperse}$ is 2.5 minutes and a ROD of U* of 11.9%, t_{ROD} of 2.23 and n is 1.15.

Having thus described the invention in detail, it will be obvious to those skilled in the art that various changes may be made without departing from the scope of the invention and the invention is not to be considered limited to what is described in the specification.

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WHAT IS CLAIMED IS:

1.   A granular detergent composition having an average bulk density of at least about 400 g/L and characterized by a rate of dispersion as defined by the equation:

$$R = R^* + (1 - R^*)\exp\left(-\left(\frac{t}{DT(t_{wash})}\right)^m\right)$$

where R is the residual undissolved detergent at any point in time, t, R* is the long term residual undispersed detergent having a value of less than about 14% of the total amount of an initial dosage of detergent, t is any single point in time, m is a stretching exponent having a value of less than about 2, DT is dispersion time having a value of less than about 0.5 and t_{wash} is the time of the wash cycle.

2.   The granular detergent composition as claimed in Claim 1 wherein at least 90% of the insoluble residues of the granular detergent composition have an average particle size of less than about 10 μm.

3.   The granular detergent composition as claimed in Claim 1 wherein R* has a value of less than about 7%, m has a value of less than about 1.5 and DT has a value of less than about 0.25.

4.   The granular detergent composition as claimed in Claim 3 wherein R* has a value of less than about 3.5%, m has a value of less than about 1 and DT has a value of less than about 0.12.

5.   The granular detergent composition as claimed in Claim 1 wherein said detergent composition has a rate of dissolution as defined by the equation:

$$U = U^* + (1 - U^*)\exp\left(-\left(\frac{t}{RT(t_{wash})}\right)^n\right)$$

where U is the fraction of undissolved surfactant at any point in time, t, U* is the long term surfactant residual undissolved surfactant having a value of less than about 14% of the total amount of an initial dosage of surfactant, t is any single point in time, n is a stretching exponent having a value of less than about 2, RT is dissolution time having a value of less than about 0.5 and t_{wash} is the time of the wash cycle.

6. The granular detergent composition as claimed in Claim 4 wherein U* has a value of less than about 7%, n has a value of less than about 1.5 and RT has a value of less than about 0.25.

7. The granular detergent composition as claimed in Claim 5 wherein U* has a value of less than about 3.5%, n has a value of less than about 1 and RT has a value of less than about 0.12.

8. The composition as claimed in any of claims 1-7 wherein said composition has insoluble residues and at least about 90%of said insoluble residues have a particle size of less than 15 μm.

As a below named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated below next to my name.

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled __Granular Detergent Compositions Having Improved Solubility__
the specification of which

(check   []   is attached hereto.
one)   [X]   was filed on ___September 24, 1999___ as
PCT International Application Serial No. ___PCT/US99/22396___
and was amended on _____
(if applicable)

I hereby state that I have reviewed and understand the contents of the above identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose information which is material to patentability as defined in Title 37 Code of Federal Regulations §1.56.

I hereby claim foreign priority benefits under Title 35 United States Code §119(a)-(d) or §365(b) of any foreign application(s) for patent or inventor's certificate, or §365(a) of any PCT International application which designated at least one country other than the United States of America, listed below and have also identified below any foreign application for patent or inventor's certificate, or of any PCT international application having a filing date before that of the application on which priority is claimed:

Prior Foreign Application(s)   Priority Claimed

(Number)	(Country)	(Day/Month/Year Filed)	[] Yes	[] No

I hereby claim the benefit under Title 35, United States Code §119(e) of any United States provisional application(s) listed below.

___60/148,258___	___August 11, 1999___	_____	_____
Application Serial No.	Filing Date	Application Serial No.	Filing Date

I hereby claim the benefit under Title 35 United States Code §120 of any United States application(s), or §365(c) of any PCT International application designating the United States of America, listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States or PCT International application in the manner provided by the first paragraph of Title 35 United States Code §112, I acknowledge the duty to disclose information which is material to patentability as defined in Title 37 Code of Federal Regulations §1.56 which became available between the filing date of the prior application and the national or PCT international filing date of this application:

U.S. Parent Application Number	PCT Parent Number	Parent Filing Date (MM/DD/YYYY)	Parent Patent Number (If applicable)
	PCT/US98/20223	September 25, 1998	

As named inventor, I hereby appoint the following registered practitioner(s) to prosecute this application and transact all business in the Patent and Trademark Office connected therewith:

Atty Name	Atty Reg Number.	Associate Power of Attorney Attached [ ] Yes [X] No
Brian M. Bolam	37,513	
C. Brant Cook	39,151	
Marianne Dressman	42,498	
Steven W. Miller	31,984	
T. David Reed	32,931	
Timothy B. Guffey	41,048	

SEND CORRESPONDENCE TO:

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I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Full name of sole or first joint inventor _Paur R. Mort, III_

Inventor's signature _____   3/30/2001
                                                              Date

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Inventor's signature _____   3/27/01
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                                                              Date

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Inventor's signature _____
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Citizenship _Great Britian_

Post Office Address _81, Windburgh Drive; Southfield Lea, Cramlington Northumberland; England NE23 6NT_

7713dec

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Full name of fourth joint inventor, if any Kevin Todd Norwood
Inventor's signature _____

Date _____
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Full name of fifth joint inventor, if any George (nmn) Burgess
Inventor's signature _____
Date 4th July, 2001
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Citizenship Great Britian
Post Office Address 81, Windburgh Drive; Southfield Lea, Cramlington Northumberland; England NE23 6NT

7713dec

DECLARATION COMBINED WITH POWER OF ATTORNEY

As a below named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated below next to my name.

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled _Granular Detergent Compositions Having Improved Solubility_
the specification of which

| (check | [] | is attached hereto. |
| one) | [X] | was filed on ___September 24, 1999___ as |

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Prior Foreign Application(s)   Priority Claimed
                                                                []   []
(Number)   (Country)   (Day/Month/Year Filed)   Yes   No

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| 60/148,258 | August 11, 1999 | | |
| Application Serial No. | Filing Date | Application Serial No. | Filing Date |

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	PCT/US98/20223	September 25, 1998	

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C. Brant Cook	39,151	
Marianne Dressman	42,498	
Steven W. Miller	31,984	
T. David Reed	32,931	
Timothy B. Guffey	41,048	

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IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In the U.S. National Phase Entry
Under 35 USC 371 from
International Application of
CAPECI, Scott William et al.
Int'l. Application No. PCT/US99/22396
Filed in the RO/US on 24 September 1999
Entitled: *Granular Detergent Compositions*
         Having Improved Solubility Profiles

ASSOCIATE POWER OF ATTORNEY

Assistant Commissioner for Patents
Box PCT
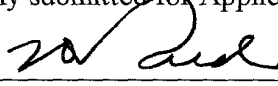Washington, D.C. 20231

Dear Sir:

You are requested to recognize K. W. Zerby (Registration No. 32,323), B. M. Bolam (Registration No. 37,513), C. B. Cook (Registration No. 39,151), F. C. Turner (Registration No. 39,863), M. Dressman (Registration No. 42,498), and R. S. Echler, Sr. (Registration No. 41,006) of The Procter & Gamble Company, Cincinnati, Ohio, as Associate Attorneys to prosecute this application, to make alterations and amendments therein, and to transact all business in the Patent Office connected with the application or with the patent granted thereupon.

Please address all future communications to:

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          Respectfully submitted for Applicants,

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